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Processing of B₄C Particulate Reinforced 6061 Aluminum Matrix Composites by melt stirring involving two-step addition

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Abstract

In the present work, 11wt% B₄C particulate reinforced 6061 Al matrix composites were produced by conventional melt stirring method. Processing of composite is carried out at a temperature of 750°C involving two stage additions. Preheated B₄C particles along with K₂TiF₆ halide salt (with ratio of 0.3) was introduced in steps of two rather than adding all at once. Characterization of the prepared composites is done using SEM/EDX and X-RD studies. Fairly uniform distribution of B₄C particulates without clustering in 6061Al matrix was evident from SEM studies. The prepared composite consists of α -Al, B₄C and minor phases like Al₃Ti, AlB₂ and Al₃BC are confirmed by XRD studies. The addition of B₄C particulates to 6061Al matrix has resulted in improvements in mechanical properties of the base alloy.

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Keywords: Melt stirring; B₄C; K₂TiF₆ halide salt; two step addition; microstructure; mechanical properties;

1. Introduction

With the increase in demand for less denser and high stiffer component, Al matrix composite find its place in the area of aerospace and non-aerospace categories. The replacement of the nickel cast iron in conventional diesel engine piston crown by aluminum matrix composite has resulted in a lighter, more abrasive and cheaper product [K. K. Chawla (1998)]. In applications such as automotive drive shafts, cylinder liners, connecting rods and because of low thermal expansion and conductivity Al based composites are used as heat sinks in chip carrier multilayer boards, high speed integrated circuit packages for computers and in base plates for electronic equipments [K. K. Chawla (1998)]. Aluminium matrix being lighter can be strengthened by reinforcing less dense hard ceramic particles such as SiC, Al₂O₃, TiB₂, B₄C etc which shows improvements in properties [Ramesh et al. (2009) and

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Lashgari et al. (2010)]. Number of fabrication technique are being employed for production of MMC's like solid state methods, liquid state method, deposition and insitu process [William et al. (1998) and Kalaiselvan et al. (2011)] out of which liquid state methods in particular stir casting has an attractive economic aspect combined with wide selection of materials and processing conditions [Shorowordi et al. (2003) and Kerti et al. (2008)]. In traditional stir casting process, reinforcement material is being added to molten matrix and poured in to permanent molds after stirring mechanism [Gopalkrishnan et al. (2010)]. Stir casting process enhances better bonding between matrix and reinforcement because of the stirring action. The main concern in fabricating MMC's is difficulty in achieving homogeneous distribution of particles, wettability, chemical reactions at the interface and porosity [Hashim et al. (1997), Hashim et al. (2002) and Hashim et al. (2002)].

Most of research work has been dedicated to fabrication of Al based MMC's being SiC, Al₂O₃, TiB₂ as reinforcement material; but use of boron carbide particulate is very limited due to high cost and poor wetting with the Al matrix below 1100°C [Canacki et al. (2007)]. B₄C is having hardness of 3800Hv considered as third hardest material next to diamond and C-boron nitride with good impact and wear resistance, low specific density (2.52g/cc), low thermal conductivity (35W/mK) and high stiffness (445GPa) which makes it to find applications like ballistic armor, as abrasive, nozzles etc [Katkar et al. (2011)]. Wettability of ceramic particles can be improved by several ways which include pre-treatment, use of surface active agents [Kennedy et al. (2001)] which decreases surface tension and interfacial forces [Sajjadi et al. (2011)]. Coating of B₄C particles with Ti powder results in formation of complex surface layers of TiB and TiC [Mogilevsky et al. (1995)] and these interfacial products have greater metallic character to their bonding increasing better incorporation of B₄C particles into melt. Use of K₂TiF₆ halide salt during casting is another method being practiced which has resulted in improved bonding between Al and B₄C particles facilitating better mechanical properties in the composite material [Kalaiselvan et al. (2011)]. Further, during melt stirring, getting proper distribution of reinforcing particles in the matrix is challenging. Because after wetting the particles sink or float due to density differences as a consequence, the dispersion becomes non homogeneous which may lead to clustering and segregation of particles at a particular place in the melt. Such effect could lead to several microstructural defects like porosities, oxide inclusions and interfacial reactions [Sajjadi et al. (2011)].

In the present works an attempt is being made to process 6061Al- B₄C particulate composites at a temperature of 750°C (less than 800°C) by melt stirring involving two step additions. The two step addition method used in the present work helps to overcome the problem of agglomeration/clustering of reinforcing particles. During composite preparation, pre-heated mix containing B₄C particles and K₂TiF₆ flux was added in steps of two into melt to enhance wetting and incorporation of B₄C particles into molten Al. Further, the composites prepared were subjected to evaluation of mechanical properties to note the extent of improvement achieved thereby.

2. Experimental Details

B₄C particle reinforced 6061Al alloy composites containing 11wt% of B₄Cp were produced by melt stirring using two step additions. The chemical composition of the 6061Al alloy matrix was analyzed with the help of Atomic Absorption Spectroscopy (VARIAN) and is presented in Table 1. Particulates of the B₄C having mean size of 88µm are used as reinforcing materials. Kennedy et al. (2001) in their work mentioned that, the wettability of B₄C_p by the liquid matrix is poor at temperatures below 1000°C. Therefore, in order to improve wettability of B₄C particulates by the liquid matrix at temperatures below 1000°C, halide salt of hexafluorotitanate (K₂TiF₆) is utilized. Further, to achieve 100% yield, Ti/B₄C ratio should be equal to 0.07 at least [Isilkerti et al. (2010) and Kennedy A. R et al. (2001)]. However, in the current investigation an attempt is made to synthesize composite at temperature of 750°C, hence Ti/B₄C ratio of 0.3 was applied. The B₄C particulates were mixed with hexafluorotitanate (K₂TiF₆) in the ratio of 0.3 and this mixture were preheated to a temperature of 250°C before introducing into the melt. A batch of 500gm was charged in a graphite crucible and melting was carried out at a temperature of 750°C in an electric furnace under the cover flux. Careful control of temperature was done to an accuracy of ±5°C using digital temperature controller. Cover flux added will result in decrease of contact angle and surface tension forces, thereby helps in improving the wettability of B₄C particles at a lower temperature by the matrix. Once the required temperature was achieved,

degassing was carried out using solid hexachloroethane (C_2Cl_6) to release all the absorbed gases. The stirring of the melt was done with the help of a Zirconia coated steel rod to generate vortex [Hashim et al. (1999)]. A spindle speed of 250 RPM and stirring time 5-8 min., were adopted. Through this vortex, a preheated mixture of B_4C and K_2TiF_6 salt were introduced in steps of two at a constant feed rate of 1.2-1.4 g/Sec. Two step additions involve dividing the entire weight of the mixture (B_4C and K_2TiF_6) into two equal weights and then individual weights are added to the melt in two steps rather than adding all at once. At every stage, stirring was carried out before and after insertion of the mixture to avoid agglomeration and separation of particles and to assure uniform distribution of reinforcing particles in the melt. After holding the melt for a period of 5 min, the melt was poured from $750^\circ C$ in to a preheated metallic mould having dimensions of 125mm length x 25mm diameter.

2.1. Microstructural Characterization and Mechanical Testing

The prepared composites were subjected to microstructural characterization using Scanning Electron Microscopy equipped with EDX analysis (Hitachi Su-1500 model) to identify morphology and distribution of B_4C particles in 6061Al matrix. XRD analysis of the prepared composite was done using XRD Machine-7000; M/s Shimadzu Analytical India Pvt. Ltd. The experimental density of the prepared composite was determined using Archimedes's principle and compared with the theoretical densities of 6061Al alloy (2.7 g/cm^3) and B_4C particles (2.51 g/cm^3). Micro hardness measurements were conducted on prepared composite at 30 different locations on the same specimen using micro-Vickers hardness tester (ZWICK Tester) under a load of 2N with dwell time of 10 Sec. The mechanical properties of the prepared composites were measured under tension using a Computerized Universal Testing Machine (INSTRON) as per ASTM E08-8 standards (2004). Tensile tests were conducted on three specimens for each composition and average value is reported.

Table 1: Shows the chemical composition of the 6061Al alloy used in the present study

Elements	Mg	Si	Fe	Cu	Mn	Cr	Zn	Ti	Al
% by Weight	0.95	0.54	0.22	0.17	0.13	0.09	0.08	0.01	Bal

3. Results and Discussions

1 Characterization using SEM/EDX

Microstructural characterization of the 6061Al alloy reinforced with 11wt% B_4C particulates is carried out with the help of Scanning Electron Microscope and are presented in Fig. 1 (a-e). Fig. 1 (a) shows SEM microphotograph of 6061Al-11wt% B_4C_p composite without addition of K_2TiF_6 salt and reveals completely absence of B_4C particles. The complete absence of B_4C particles clearly suggests that the liquid Al matrix has poor wettability of B_4C particle at temperature below $1000^\circ C$ [Canakci et al. (2007) and Sajjadi et al. (2011)]. Fig. 1 (b) shows SEM microphotograph of the same composite prepared with the addition of preheated mixture containing K_2TiF_6 halide salt (ratio 0.3) and B_4C particle in single step. Microphotographs clearly revealed the presence of B_4C particles in Al matrix. i. e. Addition of K_2TiF_6 salt has improved wettability of B_4C particles by liquid Al matrix at pouring temperature of $750^\circ C$. It is believed that the presence of titanium in the form of K_2TiF_6 halide salt contributes towards removal of the oxide layer from Al melt thereby improving the wettability [Baradeswaran et al. (2013)]. However, the distribution of the particles in matrix is not homogeneous instead clustering of the particles is seen. Also matrix contains several regions which are devoid of B_4C particles. Fig. 1 (c) shows SEM microphotographs of 6061Al-11wt% B_4C_p composite prepared with two step addition of preheated mixture. It is clear that two step addition has resulted in fairly homogeneous distribution of B_4C reinforcing particles in the Al matrix. Also no clustering or agglomerations of the particles were seen throughout the specimen which could be due to better stirring action achieved via two step addition along with K_2TiF_6 salt. Kalaiselvan et al. in their work on Al- B_4C MMC's have used processing temperature of $800^\circ C$ considering the poor wettability of B_4C by Al-matrix. However, in the present work a temperature of $750^\circ C$ combined with preheating of the mixture (K_2TiF_6 salt + B_4C_p) and two step addition has resulted in better wettability and dispersion of B_4C particles. The possible explanation for improved wettability is; according to Jonas (2001) the heat of reaction between Al and K_2TiF_6 salt at $800^\circ C$ is $-8.3 \times 10^{-5} \text{ J/Mol}$, i.e the reaction between molten Al and K_2TiF_6 is more intense and exothermic in nature. In the present work K_2TiF_6 salt is

used in the ratio of 0.3 as a result more amount of salt is available for the exothermic reaction, which may have resulted in temperature rise beyond 800°C, thereby improved wettability. SEM microphotographs have also revealed the presence of the white Titanium layer surrounding B₄C particles as shown in Fig.1 (d). In order to confirm the presence of Ti compound layer EDX analysis were carried out at the edge of the B₄C particle which is adhered in the form of white layer surrounding B₄C particle and is reported in Fig.1 (e). The K₂TiF₆ salt undergoes exothermic reaction when comes in contact with the liquid Al matrix. The reaction being vigorous and exothermic in nature thereby leads to formation of the possibly Al₃Ti compound layer as evident from EDX analysis. The exothermic nature of reaction between K₂TiF₆ salt and liquid Al which lead to formation of Al₃Ti is governed by the following equation [Auradi et al. (2008)]



2 XRD studies

In order to identify the various phases formed during the production of 6061Al-11wt%B₄C_p composite XRD analysis was carried out and the results are presented in Fig. 2. XRD results have shown the intensity peaks corresponding to the phases of α-Al, B₄C, Al₃Ti, AlB₂ and Al₃BC. Presence of Al₃Ti phase is clear indication of Ti compound/layer that was formed around the B₄C particles due to the reaction between K₂TiF₆ salt and liquid Al matrix. XRD analysis has also revealed the peaks of AlB₂ and Al₃BC, which are the products formed at the interface between Al and B₄C particle. It has been reported that within a temperature range of 627-1000°C, B₄C undergoes interaction with solid or liquid aluminium [Shorowadi et al. (2003), Viala et al. (1997)]. The reaction result in formation of Al₃BC and AlB₂ up to a temperature of 868°C and at temperatures higher than 868°C, Al₃BC is still formed while Al₃B₄₈C₂ replaces AlB₂. Since the present work involves synthesizing 6061Al-B₄C particulate composites at a lower temperature of 750°C, the expected phases would be AlB₂ and Al₃BC and the same were reflected in XRD studies. Further, the low intensities of AlB₂ and Al₃BC peaks suggests not all the B₄C particles are involved in the chemical interaction with Al. Probably the particles which are uncovered by the Ti layer might be contributing towards the formation of AlB₂ and Al₃BC. Therefore, detailed interfacial studies are necessary in order to fully understand the chemical interaction products formed at the Al-B₄C interface.

3 Evaluation of physical and mechanical properties

3.1 Density and Hardness

The density measurement was done using water displacement technique (Archimedes's Principle). The results of density measurements of the matrix and reinforcements are represented in Fig. 3(a) and (b). Rule of mixture is being used to predict theoretical density and percentage porosity was calculated based on the theoretical density of the samples [Canakci et al. (2011) and Topcu et al.(2009)]. From Fig. 3(a) it is clear that measured density of the composite is lower than that of the base 6061Al matrix. The decreases in density of composite obtained in the present work are in good agreement with earlier works of Mohanty et al. (2008) and Abdullaha et. al (2012). The decrease in density is as expected because B₄C particle has a lower density (2.52g/cm³) when compared to that of Al. Therefore, addition of less denser material to a high density matrix, decreases overall density of the composite. Further, improved wettability of B₄C particle by liquid Al achieved with the addition of salt introducing more and more amount of B₄C into melt thereby the density of overall composite decreases [Kok (2005)]. Further, the presence of pores can be attributed to factors like gas entrapment during stirring, hydrogen evolution, pouring distance from crucible to mold and shrinkage during solidification [Sagjjadi et al. (2011) and Hashim et al. (1999)].

Fig.3 (b) shows the comparison between micro hardness values of 6061 matrix and the composite prepared. It is clear from the results that 6061Al-11wt%B₄C_p composite has shown higher hardness when compared to the matrix. The Vickers hardness of B₄C is 38GPa [Katkar et al. (2011)] which is very high when compared to that of 6061Al and it is believed that addition of harder materials to a softer matrix lead to increase in overall hardness of the composite. Further, the increase in hardness due to the addition of B₄C particles can be attributed to uniform

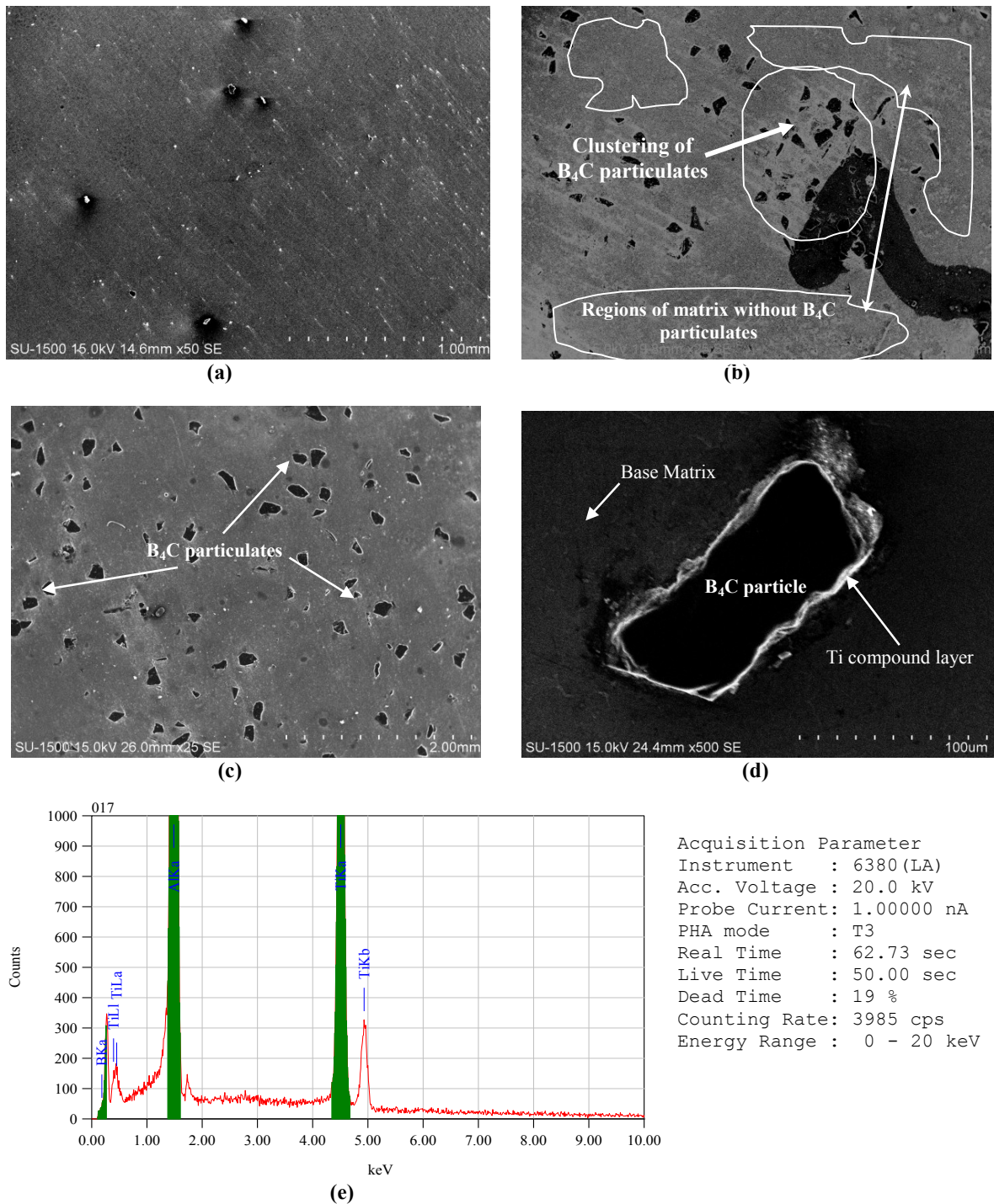


Fig.1 a-e: Shows SEM microphotographs of 6061Al with 11 wt% B₄C Composites prepared using novel two step addition at 750^oC (a) 6061Al with 11wt% B₄C but without K₂TiF₆ salt (b) 6061Al + 11wt% B₄C + K₂TiF₆ salt but with single step addition (c) 6061Al + 11wt% B₄C + K₂TiF₆ salt with two step addition (d) higher magnification SEM microphotograph around the B₄C particle showing the presence of white layer of Ti compound (e) EDX spectrum taken on the edge of the B₄C particle.

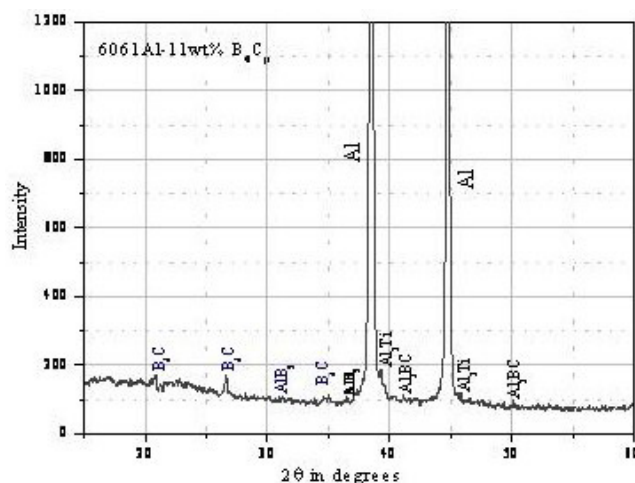


Fig. 2 X-ray diffraction pattern of 6061Al-11wt%B₄C composite prepared at 750°C via melt stirring method with two step addition of B₄C particulates

distribution of B₄C particulates in Al matrix leading to particulate strengthening effect of the matrix alloy. It has been reported that the presence of hard reinforcement particles offer resistance to plastic deformation and due to the increased strain energy the hardness of the composites is increased at the peripheral of the particles dispersed in the matrix [Ramesh et al. (2009), Baradeshwaran et al. (2013) and Mazaheri et al. (2013)].

3.2 Evaluation of Mechanical Properties

The mechanical properties such as yield stress, ultimate tensile strength and % elongation were evaluated both on 6061Al and 6061Al reinforced with 11wt% B₄C particulate composites. Results of the same are presented in Fig. 3(c). It is clear from Fig. 3(c) that mechanical properties of the 6061Al matrix are improved with the addition of B₄C particulates when compared to that of base matrix alone. The extent of improvement obtained in the 6061Al alloy after addition of 11wt% of B₄C particles were 44.35% (yield stress) and 42.6% (ultimate tensile strength) respectively. On the other hand the ductility of the composites decreases with addition of B₄C particulates which could be due to the fact that presence of the B₄C particles causes strain hardening during plastic deformation, thereby decreasing the ductility [Alizadeh et al. (2010)]. In addition, the presence of porosities (mainly in the interface of B₄C particles and metal matrix) and interfacial products formed at the interface could be other reasons for lower ductility in the composite specimens. The improvements in mechanical properties observed with composites over 6061Al matrix could be due to two factors. Firstly, presence of small second phase particles uniformly distributed in a ductile matrix are considered to be the common source of alloy strengthening. Secondly, the addition of B₄C particles to the Al matrix has resulted in decrease in grain size of the matrix alloy. The grain size can be related to yield stress (other mechanical properties) according to Hall Petch equation [Sekine et al. (1995)].

$$\sigma_0 = \sigma_i + K D^{-1/2} \text{ -----(2)}$$

where σ_0 , σ_i , K and D are yield stress, friction stress, locking parameter and grain diameter, respectively. According to equation (2) as grain diameter (D) decreases the yield stress of the material increases. In the present work, it is thought that B₄C particles, when added to Al melt act as heterogeneous nucleating sites during solidification and thereby lead to decrease in grain size and thereby improvements in mechanical properties. The results and trends obtained in the present work are in good agreement with those of earlier researchers [Faith et al (2010), Kalaiselvan et al. (2011), Shi et al. (2011) and Abdullah et al. (2012)].

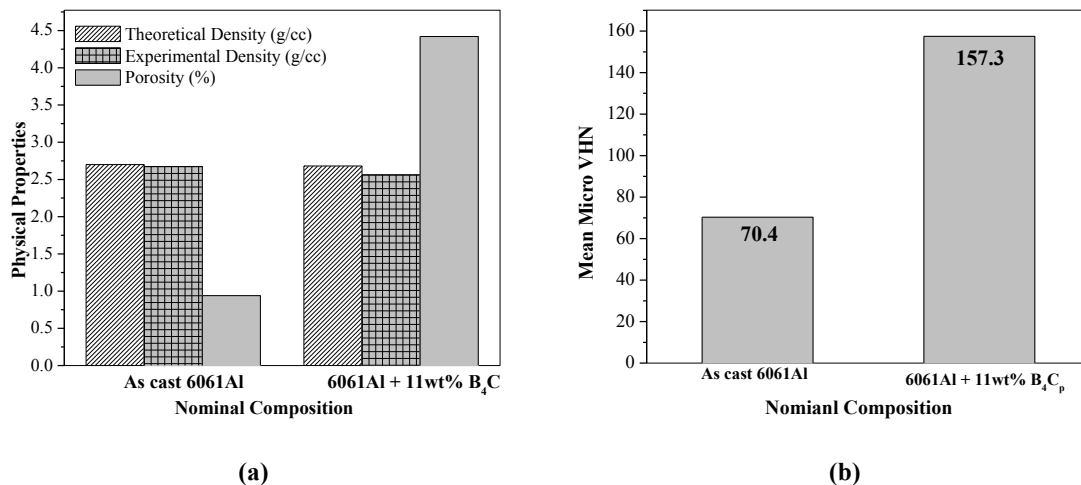
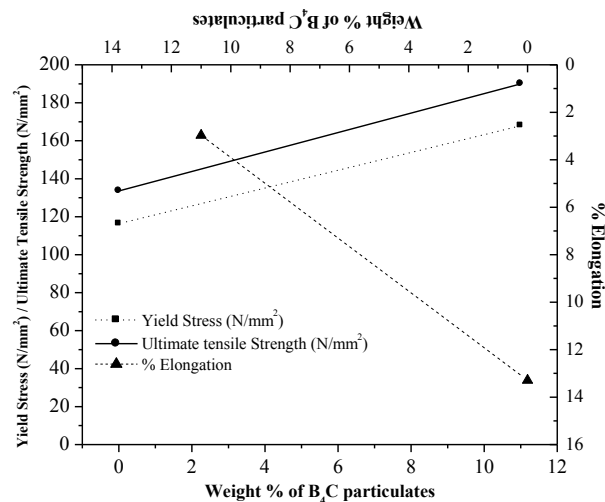


Fig. 3 shows variations in (a) density and porosity (b) hardness of 6061Al matrix before and after addition of 11wt% of B₄C particulates prepared at 750°C using two step addition melt stirring method.



Shows variations in mechanical properties (Yield Stress, UTS and % Elongation) of 6061Al matrix before and after addition of 11wt% of B₄C particulates prepared at 750°C using two step addition melt stirring method.

4. Conclusions

The present work on processing of 6061Al reinforced 11wt% B₄C particulate composite by two step addition via melt stirring method has contributed to the following conclusions.

1. 6061Al alloy reinforced with 11wt% B₄C particulate composites were successfully produced at temperature of 750°C via melt stirring involving two step addition method.
2. Two step addition method combined with preheating of the mixture (K₂TiF₆ + B₄C_p) during melt stirring has resulted in improved wettability and better dispersion of B₄C particulates in 6061Al matrix when compared to the single step addition.

3. XRD analysis confirmed the presence of α -Al, B_4C and minor phases of Al_3Ti , AlB_2 and Al_3BC in the composite prepared.
4. The addition of B_4C particulates to the 6061Al matrix has led to improved mechanical properties (Hardness, Yield Stress, UTS) when compared to the matrix alone whereas ductility decreases. The extent of improvements obtained in Yield Stress and Ultimate Tensile Strength were 44.35% and 42.6% respectively.

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